PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6:		(11) International Publication Number:	. WO 98/30326
B01J 20/28, B01D 39/00	A1	(43) International Publication Date:	16 July 1998 (16.07.98)

(21) International Application Number:	PCT/GB98/00001	(81) Designated States: CA, CN, GB, JP, US, European par
(22)		BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, I

(22) International Filing Date:	9 January 1998 (09.01.98)

(30) Priority Data:		
9700339.6	9 January 1997 (09.01.97)	GB
9725991.5	9 December 1997 (09.12.97)	GB
9725993.1	9 December 1997 (09.12.97)	GB

(71) Applicant (for all designated States except US): BONDED FI-BRE FABRIC LIMITED [GB/GB]; Bath Road, Bridgewater, Somerset TA6 4NZ (GB).

(72) Inventors; and (75) Inventors/Applicants (for US only): ABERCROMBIE, Archibald, John [GB/GB]; Redmier, Maunsel Road, North Pertherton, Bridgwater, Somerset TA7 0BP (GB). MAAN, Ranber [GB/GB]; 6 Badgers Close, Bishops Hull, Taunton TA1 5HL (GB). BATTYE, Anthony, George [GB/GB]; 68 Moorland Road, Bridgwater, Somerset TA6 4JS (GB).

(74) Agent: FITZPATRICKS; 4 West Regent Street, Glasgow G2 1RS (GB).

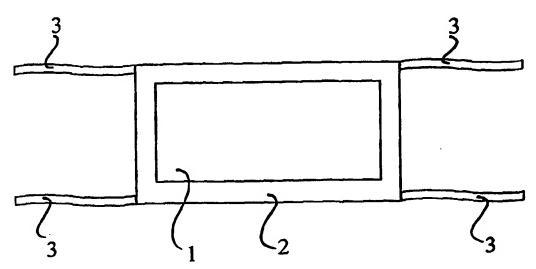
atent (AT, LU, MC, NL, PT, SE).

Published

With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: FILTER MEDIA



(57) Abstract

An adsorbent media, particularly of a type formed from a sheet-form carrier substrate, incorporating particulate adsorbents therein for use filtration of fluids, i.e., by gas/solid liquid/solid interactions wherein the contaminant atom, ion, or molecule of gas or liquid adheres or is otherwise retained by the solid adsorbent particle thereby removing it as a contaminant from the filtered fluid is provided by forming a fluid-permeable carrier substrate containing fibres capable of withstanding prolonged contact with a fluid to be filtered, treating the carrier substrate to introduce fixing agents including a binder and a film-former, and particulate adsorbent material(s) to the carrier substrate, wherein the amount of fixing agents, particularly of film former is just sufficient to retain the adsorbent material(s) in the carrier substrate whereby shedding of the adsorbent therefrom is substantially avoided.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
ΑU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of Americ
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
RE	Estonia	LR	Liberia	SG	Singapore		

FILTER MEDIA

1

present invention relates to adsorbent media. The particularly of a type formed from a sheet-form carrier substrate incorporating particulate adsorbents therein, and uses thereof in filtration of fluids i.e. by gas/solid liquid/solid interactions wherein the contaminant atom, ion, or molecule of gas or liquid adheres or is otherwise retained by the solid adsorbent particle thereby removing it as a contaminant from the filtered fluid. The invention thus finds application in the adsorption of toxic, odiferous, nuisance, radioactive or irritant chemicals from a gaseous or vapour atmosphere, or from a body of liquid, particularly, but not exclusively, in removal of dissolved organic substances from aqueous liquids for example for personal protection.

5

10

25

30

Known adsorbent materials include activated carbon, zeolites, silica and metal oxides including alumina which are often used in particulate form for the purposes of maximising the adsorbent surface. EP-A-O 432 438 describes a deodorising unit for air cleaners and air conditioners formed from a moulded mixture of such adsorbent particles, fine plastics particles and a reinforcing fibre.

A water permeable container filled with a granular adsorbent is described in JP-A-54 105 851, for use in treatment of sewage, industrial effluent etc., containing harmful substances such as heavy metals, N compounds, phosphates etc. The granular adsorbents mentioned for that purpose include activated carbon, ion exchange resins, chelate resins, calcium silicate, powdered shell, zeolites, coal, activated alumina, magnesium aluminate, and porous synthetic resins.

An adsorption filter element comprising a granular adsorbent, such as active carbon, zeolite, silica gel and stone, bonded using a hydrophilic binder e.g. acrylate synthetic resin, covered with a mesh spacer (opening less than 10 mm, thickness 0.2-5 mm) and laminated with filter sheet, e.g. woven cloth and non-woven cloth is described in JP-A-82 94 609.

A protective mask is described in US-A-4 643 182 which is formed from a porous non-woven sheet material which is made of glass fibres and a suitable binding agent such as polyvinyl alcohol, and which may optionally contain a gas adsorbent substance. The mask is intended to protect a wearer from the effects of toxic gases and the like. The glass fibres are from about 0.5 microns to 4 microns in diameter and may be from 0.1 inches to 0.25 inches in length. The gas adsorbent particles described there include activated carbon particles, crystalline molecular sieve particles or zeolite particles, and the mask may also include anion exchange resin particles.

10

15

It is known to make an activated carbon filtration medium

by spraying a liquid or foam containing activated carbon

particles onto a substrate. An advantage of this process is

that the substrate dries quickly thus speeding up the

manufacturing process. In addition, this process results in a

large proportion of the free surface area of the activated

carbon particles being available for chemical adsorption.

However, a disadvantage of this process is that the activated

carbon particles are poorly held on or within the substrate

and as a result there is a danger of said particles being shed

during normal use.

30 It is also known to manufacture an activated carbon

15

20

3

PCT/GB98/00001

filtration medium by a liquid or foam immersion process whereby a substrate is immersed in a liquid or foam containing particles. This process allows activated carbon different types of substrate to be impregnated with activated carbon particles, said substrates being chosen according to the atmosphere to which the filtration medium is to be exposed It also allows for a more even dispersion of activated carbon particles within the matrix of the substrate. in order to ensure that the activated carbon particles have a sufficient surface area available for chemical adsorption of impurities, the activated carbon particles are held generally by entrapment within the matrix of the substrate. Thus, said activated carbon particles are poorly held within the substrate matrix and are easily shed during normal use.

Activated carbon filtration media can, for example, be employed to adsorb toxic, odoriferous, nuisance, radioactive or irritant chemicals from a gaseous atmosphere or to adsorb dissolved organic compounds from a liquid atmosphere. However, the problem of shedding of the activated carbon particles from the substrate of a filtration medium can lead to the failure of the medium and can in itself represent a hazard particularly where the medium is incorporated in devices or garments for the protection of personnel.

Shedding is undesirable with any adsorbent material but is a problem which is particularly acute with regard to activated carbon due to the blackening of surfaces contacted by the shed particles. Thus handling of filtration media during assembly, maintenance operations etc. is unpleasant and requires clean-up operations and other precautions to avoid contamination with the shed materials. Often such problems are addressed by

use of binders which can reduce the effective surface available for adsorbent activity.

An object of the present invention is to obviate or mitigate the aforesaid problems. In particular it is an object of at least one aspect of this invention to address the shedding problem. Another object of the invention is to provide improved filtration media suitable for general use in fluid purification by adsorption of impurities from gaseous and/or vapour atmospheres, or from a body of liquid. A further object is to provide flexible adsorbent materials which may be readily adopted in protective apparel. A still further object is to provide an effective manufacturing process for producing such adsorbent materials and filtration media (hereinafter, simply "filter media").

10

25

30

According to a first aspect of the invention filter media comprises a fibrous material, preferably a dry laid fibrous web or the like flexible substrate incorporating an adsorbent material in particulate form wherein the adsorbent material is retained within the filter media using a fixing agent whereby in use shedding of the adsorbent material therefrom is inhibited.

The fibrous material is preferably provided as a non-woven fabric, preferably as a dry laid fibrous web or the like flexible substrate. Alternatively, the substrate may comprise a woven fabric, a scrim fabric or any other suitable substrate having a structure capable of retaining adsorbent particles.

Additional fluid-permeable fabrics may be incorporated in the filter media, and may be of a different structure to the fibrous material incorporating the adsorbent material. The additional fluid permeable fabrics may incorporate different **WO 98/30326** 5

15

20

25

30

adsorbent materials. The filter media may be formed as a flat flexible sheet which may be adapted to conform to a surface, e.g. for the purposes of inclusion in a garment, as a laminate, a scroll made up of such a sheet or laminate, or a wadding.

PCT/GB98/00001

According to another aspect of the invention there is provided a process for forming a filter media comprising providing a fluid-permeable carrier substrate containing fibres which are selected from fibres capable of withstanding prolonged contact with a fluid to be filtered, treating the carrier substrate to introduce fixing agents including a a film-former, and particulate binder and adsorbent material(s) to the carrier substrate, wherein the amount of fixing agents, is just sufficient to retain the adsorbent material(s) in the carrier substrate whereby shedding of the adsorbent therefrom is substantially avoided.

Preferably the filter media is formed by selecting a fluid-permeable carrier substrate consisting essentially of fibres and impregnating the substrate with a liquid containing an adsorbent material in particulate form and fixing agents for promoting particle retention in the substrate without occluding the permeability of the substrate, and separating the impregnated substrate and the liquid after a predetermined period sufficient to obtain retention of the particulate adsorbent material in the substrate.

A preferred liquid for imparting the desired non-shedding characteristics contains a latex binder, such as an acrylics or styrene butadiene latex or natural rubber based binder, especially containing a film former and preferably also an anti-foaming agent. The amounts are variable depending upon

6

fibres selected but a limited degree of simple experimentation is sufficient to determine the minimum amount of film former adequate to obtain a satisfactory non-shedding characteristic. The term film-former means a material capable of forming a film when dry at standard conditions of temperature and pressure (STP). Suitable materials include polyvinyl alcohol, or polyvinyl alcohol/vinyl acetate copolymers, quaternary ammonium salts of polyvinyl pyrolidine/vinyl acetate copolymer amongst other similar film formers, but polyvinyl alcohol is a preferred material.

10

15

20

25

30

Adsorbent materials may be selected according to the intended use from adsorbents which would for the purposes herein involve preferential selection from activated carbon, zeolites, inorganic compounds such as silica and metal oxides e.g. of titanium (TiO_2) , zinc (ZnO), and aluminium in crystalline or micro-crystalline or fine particulate form and mixtures of such adsorbents.

The activated carbon particles may comprise any one of charcoal, peat, coconut shell, lignite and wood, but preferably comprises charcoal or is lignite based.

Particle sizes are variable dependent upon the materials selected but a useful working range is from about 0.1 to about 300 microns, preferably up to about 75 microns, and preferably also not less than about 1 micron, more preferably not less than 25 microns.

The carrier substrate material may be a non-woven fibre fabric or similar sheet or mat capable of being impregnated with an adsorbent-containing liquid. The impregnation may be accomplished by a number of methods which would seek to achieve saturation of the fibres to obtain uniform

distribution of the adsorbent and fixing agents (treatment liquor) throughout the fibrous material. These methods would include application to the fibrous material by at least one of following treatments; saturation by soaking 5 convenient manner e.g. simply delivery of the appropriate chemical treatment liquor from a hose over the fibrous material; impregnation by immersion of the fibrous material in a bath of the treatment liquor; forced impregnation into the fibrous material by application of the liquor under pressure; 10 pouring of the treatment liquor over the fibrous material by a curtain-coating device situated over a progressively advancing web of fibrous material to drench the fibrous material; spraying the treatment liquor upon the fibrous material; or an equivalent treatment of a web or mat of the fibrous substrate. 15 The method chosen will be to some extent a matter of convenience and many variants will be apparent to the skilled plant designer. An immersion treatment such as dip-coating is very flexible in that it allows simple equipment to be adopted even for a continuous production, and permits a wide variety 20 of different substrates to be handled, with the additional advantage of greater certainty of a substantially uniform dispersion of the selected adsorbent throughout the carrier substrate, provided that care is taken to ensure that the period of contact with the chemical treatment liquor is 25 adequate to ensure that the fibrous material is heavily wetted by that liquor. Thus, a batch dip bath or slow progressive advancement of a web through a dip bath may be preferred for thicker substrates. On the other hand a spray treatment permits quick drying, rapid throughput with an overall fast 30 process rate, and also facilitates presentation of more of the available free surface of the selected adsorbent. It will be recognised that a spray coating plant can offer economies in

plant volume by avoiding the need for the significant space taken up by treatment baths and saturation holding tanks. Thus a web of fibrous material of thin to medium thickness suitably suspended between spray nozzles can be saturated quickly by the pressure of the spray jets alone. Other ways of saving space for such a treatment plant would include passing a fibrous web under a header tank equipped with means in contact with the web for applying treatment liquor under pressure to forcibly impregnate the web to the desired degree of saturation. The forced penetration may be achieved or assisted by reducing ambient pressure on a surface of the web opposite to the liquor receiving surface.

10

15

In selecting the treatment method those skilled in fibre technology will naturally pay due attention to the physical characteristics of the fibrous substrate to be treated. Thus fibrous materials which may be disrupted by pressure treatments would therefore be preferentially treated by low pressure soaking treatment methods

The fibres to be used are preferably man-made durable

20 materials i.e. synthetic materials of good resilience and impervious to, or at least highly resistant to degradation under contact with the fluid(s) with which it is anticipated normal usage as a filter media would expose the fibres to. Thus one might readily consider polyesters, polyamides, and the like. Preferably one would use polyester fibres in acidic fluids, nylon or polyvinyl alcohol fibres in alkaline fluids, and rayon viscose fibres for use in contact with vegetable or mineral oil containing fluids. However these recommendations are not exhaustive and are merely illustrative of the materials contemplated for the purposes of this invention.

15

20

25

30

The fibres may be between 0.5 dtex and 6 dtex but are preferably in the range of 1 dtex to 3 dtex.

Preferably the fibres are synthetic materials formed into similar flexible sheet-form substrate, web, mat or preferably as a non-woven fabric or as a laminar composite material consisting of layers of woven and/or non-woven fibres. Non-woven fabrics suitable for the purposes of this invention are dry-laid fabrics which may be produced using a carding process known in the art such as that process particularly described in GB-A-2 151 667 to which reference is directed. However non-woven fabrics are also obtainable by entanglement processes, preferably by hydro-entanglement processes known generally in the art of non-woven fabric Such a fibre entanglement process subjects loose production. fibres or filaments to a stream or streams of jetted fluid e.g. using air streams (US-A-4 100 324) or using hydraulic fluids (US-A-3 485 706) to intermingle the fibres.

The filter media contemplated for the purposes of this invention preferably comprises a fibrous web which may contain from 20% to 90%(w/w), preferably from 30% to 60% by weight, of the filter media sheet-form substrate.

The amount of fixing agents is suitably in the range of from 5% (w/w) to 45% (w/w), but preferably is from 15% (w/w) to 30% (w/w) in terms of the finished filter media product. In relation to the treatment process, this is achievable using a liquid containing the fixing agents, which may contain from 5% (w/w) to 95% (w/w) of a latex binder, preferably from 25% (w/w) to 65% (w/w) based on the solids (dry weight as determined at STP) content of the treatment liquid. Likewise for the film former material, the content thereof in a

10

WO 98/30326 PCT/GB98/00001

treatment liquor may be from 0.05% (w/w) and 5% (w/w), with a preferred range of from 0.4% (w/w) to 1.2% (w/w). Where an antifoaming agent is used, the amount thereof can be between 0.05% (w/w) and 5.0% (w/w), the preferred range being from 1.5% (w/w) to 3.0% (w/w).

Regarding the content of adsorbent(s) this would be in the range of from 5%(w/w) and 75%(w/w), preferably from 15%(w/w) to 65%(w/w) in the finished filter media. With regard to the content of the adsorbent(s) in the treatment liquor, again based on the dry weight as determined under STP, this would be from 4%(w/w) to 94%(w/w), preferably from 1.5%(w/w) to 3.0%(w/w). Ideally the particle size thereof is in the range of 1 micron to 300 microns, preferably from 25 microns to 75 microns.

15 The treatment may be conducted by immersion in a bath of a liquid containing the selected fixing agents and selected adsorbent(s). This immersion treatment results in impregnation of the carrier substrate. Alternatively the liquid containing the selected fixing agents and selected 20 adsorbent(s) may be applied to a web of the carrier substrate by a spraying process or by a curtain coating (drench or pouron) type of application.

According to a further aspect of the invention there is provided an article of apparel, such as a protective suit,

25 face mask, industrial glove, apron or article of footwear, including a fabric comprising fibrous materials, preferably as a non-woven (e.g. dry-laid) fabric incorporating an adsorbent material in particulate form which is retained therein by means of a latex binder and a film former present in sufficient quantities to avoid shedding of the particulate

adsorbent(s) therefrom but permitting the fabric to retain a high level of fluid permeability. The advantage of such protective apparel is that the wearer may be protected against skin contact with toxic or noxious substances in the air for example.

According to a further aspect of the invention there is provided a filter cartridge for location in a filtration device comprising а non-woven fabric (e.g. dry-laid) incorporating an adsorbent material in particulate form which is retained in the fabric by means of a latex binder and a film former present in sufficient quantities to avoid shedding of the particulate adsorbent(s) therefrom but permitting the fabric to retain a high level of fluid permeability. cartridge may include conventional components in addition to at least one web, or layer, or bundle, or scroll or wadding composed of the said dry-laid non-woven fabric treated in accordance with the invention as hereinbefore described.

The invention will now be described further by way of the following illustrative examples.

20 EXAMPLE 1

5

10

15

A filter media of polyester fibres having a length in the range of 30 to 50mm and of between 1 and 3 dtex are formed into a sheet which comprises a fibrous web using the carding process detailed in GB-A-2 151 667.

25 The sheet may then be impregnated with particles of activated charcoal by a process which involves immersing said sheet for a predetermined period of time in a bath of treatment liquor in which the activated charcoal is suspended. In this particular example the activated charcoal has a particle size in the range of 25 to 75 microns.

The treatment liquor based on the dry weight as determined under conditions of standard temperature and pressure comprises 25% (w/w) of an acrylic butadiene, 2% (w/w) of a polyvinyl alcohol, and an anti-foaming agent in a suitable organic carrier medium. The bulk of the liquor is made up of the particulate activated charcoal. The anti-foaming agent is a carbon based compound.

During the immersion of the sheet in the treatment liquor the activated charcoal particles are taken up into the structure of the fibrous web and held in place by the binder and the other fixing agents used.

Once the sheet has been immersed for a sufficient period of time it is removed from the bath and the excess liquor allowed to run off and dry. The final product is the filter media.

In the filter media formed the activated charcoal remains porous to the gas or liquid passing through the filter media and also is not occluded to any significant degree by the binding agent polyvinyl alcohol/acrylic butadiene which is binding it to the fibres of the filter media.

EXAMPLE 2

10

15

20

25

A filter media of rayon viscose fibres having a length in the range of 30 to 50mm and of between 1 and 3 dtex are formed into a sheet which comprises a fibrous web using the carding process detailed in GB-A-2 151 667.

The sheet may then be impregnated with particles of activated charcoal by a process which involves immersing said sheet for a predetermined period of time in a bath of treatment liquor in which the activated charcoal is suspended.

In this particular example the activated charcoal has a particle size in the range of 150 to 175 microns.

The treatment liquor based on the dry weight as determined under conditions of standard temperature and pressure comprises 29% (w/w) of a styrene butadiene, and 1% (w/w) of a polyvinyl acetate/Vinyl acetate copolymer in a suitable organic carrier medium, and in this case no antifoaming agent is present in this case. The bulk of the liquor is made up of the particulate activated charcoal.

During the immersion of the sheet in the treatment liquor the activated charcoal particles are taken up into the structure of the fibrous web and held in place by the binder and the other fixing agents used.

Once the sheet has been immersed for a sufficient period

of time it is removed from the bath and the excess liquor

allowed to run off and dry. The final product is the filter '

media.

In the filter media formed the activated charcoal remains porous to the gas or liquid passing through the filter media and also is not occluded to any significant degree by the binding agent polyvinyl acetate/vinyl acetate copolymer/styrene butadiene which is binding it to the fibres of the filter media.

EXAMPLE 3

20

A filter media of nylon fibres having a length in the range of 30 to 50mm and of between 1 and 3 dtex are formed into a sheet which comprises a fibrous web using the carding process detailed in GB-A-2 151 667.

The sheet may then be impregnated with particles of activated charcoal by a process which involves immersing said sheet for a predetermined period of time in a bath of treatment liquor in which the activated charcoal is suspended. In this particular example the activated charcoal has a particle size in the range of 75 to 125 microns.

5

10

20

The treatment liquor based on the dry weight as determined under conditions of standard temperature and pressure comprises 35% (w/w) of a natural rubber material, 2% (w/w) of a polyvinyl alcohol, and an anti-foaming agent. The bulk of the liquid carrier material is made up of the particulate activated charcoal. The anti-foaming agent is silicon based compound.

During the immersion of the sheet in the treatment liquor

15 the activated charcoal particles are taken up into the

structure of the fibrous web and held in place by the binder

and the other fixing agents used.

Once the sheet has been immersed for a sufficient period of time it is removed from the bath and the excess liquor allowed to run off and dry. The final product is the filter media.

In the filter media formed the activated charcoal remains porous to the gas or liquid passing through the filter media and also is not occluded to any significant degree by the binding agent polyvinyl alcohol/natural rubber material which is binding it to the fibres of the filter media.

An activated carbon filtration medium not only performs the function of a chemical adsorbate in a gaseous or liquid atmosphere but also acts as a particulate filter by means of

10

15

20

25

30

15

PCT/GB98/00001

and/or electrostatic entrapment of particles physical contained in a flow of gas or liquid passing through the medium, thus providing a means of reducing the amount of impurities in the gas or liquid. The activated carbon filtration medium must remain porous to the gas or liquid to be passed therethrough. It must allow easy passage of said gas or liquid through the medium to facilitate contact of activated carbon within the medium and any impurities contained within the gas or liquid thus enabling the activated carbon, through the process of adsorption and aided by particle entrapment by the substrate structure, to reduce the amount of impurities in the gas or liquid.

An activated carbon filtration medium should exhibit some of the following features: a rapid adsorption of impurities from the gas or liquid passing through the medium; a high free surface area of activated carbon particles available for the adsorption of impurities; good fixation of the activated carbon particles in the filtration substrate in order to prevent shedding of said carbon particles under normal conditions of use; be porous to the gas or liquid to be passed through the medium; and be capable of physical and/or electrostatic entrapment of solid particles in the gas or The present invention enables these functions to be liquid. retained yet the shedding problem is solved by providing a impregnated with activated substrate which is carbon particles, wherein said particles are fixed within the structure of the substrate such that the free surface area of the carbon particles is not occluded by more than a minimum amount required for the continued efficacy of the carbon particles as an adsorbate for impurities in gaseous or liquid

PCT/GB98/00001 WO 98/30326 16

atmospheres and, in such a manner, that said filtering means does not shed the carbon particles in use.

EXAMPLE 4

A filter media is formed and treated as detailed in Example 1 except in this case the activated charcoal is 5 replaced with a particulate Zeolite.

EXAMPLE 5

A filter media is formed and treated as detailed in Example 2 except in this case the activated charcoal is replaced with particulate Titania. 10

EXAMPLE 6

A filter media is formed and treated as detailed in Example 1 except in this case the activated charcoal is replaced with a particulate Silica.

15 EXAMPLE 7

A filter media is formed and treated as detailed in Example 3 except in this case the activated charcoal is replaced with a particulate Zinc Oxide.

EXAMPLE 8

A filter media is formed and treated as detailed in 20 Example 3 except in this case the activated charcoal is replaced with a particulate Aluminium Oxide.

EXAMPLE 9

A filter media is formed from vinyl acetate fibres as per 25 example 1 and treated as per said example.

EXAMPLE 10

A filter media of polyester fibres having a length in the range of 30 to 50mm and of between 1 and 3 dtex are formed

into a sheet which comprises a fibrous web using the carding process detailed in GB-A-2 151 667.

The sheet may then be impregnated with particles of activated charcoal by a process which involves spraying the sheet a treatment liquor in which the activated charcoal is suspended, so that it is saturated from all sides and in effect immersing said sheet in the treatment liquor. In this particular example the activated charcoal has a particle size in the range of 25 to 75 microns.

The treatment liquor based on the dry weight as determined under conditions of standard temperature and pressure comprises 25% (w/w) of an acrylic butadiene, 2% (w/w) of a polyvinyl alcohol, and an anti-foaming agent in a suitable organic carrier medium. The bulk of the liquor is made up of the particulate activated charcoal. The anti-foaming agent is carbon based compound.

The effective immersion of the sheet in the treatment liquor means that the activated charcoal particles are taken up into the structure of the fibrous web and held in place by the binder and any other fixing agents used. The final product is the filter media.

In the filter media formed the activated charcoal remains porous to the gas or liquid passing through the filter media and also is not occluded to any significant degree by the binding agent polyvinyl alcohol/acrylic butadiene which is binding it to the fibres of the filter media.

EXAMPLE 11

5

20

25

A filter media of polyester fibres having a length in the range of 30 to $50\,\mathrm{mm}$ and of between 1 and 3 dtex are formed

into a sheet which comprises a fibrous web using the carding process detailed in GB-A-2 151 667.

The sheet may then be impregnated with particles of activated charcoal by a process which involves continuously dip coating the sheet in a trough of a treatment liquor in which the activated charcoal is suspended, so that it is saturated from all sides. In this particular example the activated charcoal has a particle size in the range of 25 to 75 microns.

The treatment liquor based on the dry weight as determined under conditions of standard temperature and pressure comprises 25% (w/w) of an acrylic butadiene, 2% (w/w) of a polyvinyl alcohol, and an anti-foaming agent in a suitable organic carrier medium. The bulk of the liquor is made up of the particulate activated charcoal. The anti-foaming agent is a silicon based compound.

The effective immersion of the sheet in the treatment liquor means that the activated charcoal particles are taken up into the structure of the fibrous web and held in place by the binder and any other fixing agents used.

20

The sheet is then allowed suspended so that the excess liquor is allowed to run off and the sheet is then allowed to dry. The final product is the filter media.

In the filter media formed the activated charcoal remains porous to the gas or liquid passing through the filter media and also is not occluded to any significant degree by the binding agent polyvinyl alcohol/acrylic butadiene which is binding it to the fibres of the filter media.

EXAMPLE 12

A an article of protective apparel in the form of surgical type face mask as shown in Figure 1 of the drawings and comprising a central filtering portion 1 having a surrounding edging strip 2, and securing straps 3 is formed from the filter media as detailed in Example 1. This type of mask is useful, for example to workers who are handling radioactive and /or hazardous vapour emitting chemicals such as mercury.

10 EXAMPLE 13

15

20

25

A filter media as per one aspect of the present invention and as illustrated in Figure 2 of the drawings can be made using the sheets as made and treated in accordance with the present invention. The filtering media comprises a plastic casing 10 which is designed to be push-fitted into separate face covering breathing apparatus. The casing 10 comprises a sidewall 11, a back-wall 12 having a number of perforations 12a formed therein, and a front-wall 13 formed in a lattice type way and having a series of holes therein. Once fitted in the breathing apparatus the passage of air to the user is via the holes in the front-wall 13 through the filter media 14 and out through the perforations 12a in the back-wall 12.

The filter media 14 is located and secured in the casing 10 so that any air being drawn through the filtering media has to pass therethrough before it can be breathed in by the user. The filter media 14 comprises a wad of material 15 which has a number of individual layers at least one of which layers is a sheet of material as made and treated in accordance with Example 3.

EXAMPLE 14

A filter media as per Example 11 except the wad of material 15 is with a number of layers of material made and treated in accordance with the examples detailed above. The wad can include several layers of a sheet made in accordance with one example, for example Example 1, or layers of sheets made and treated in accordance with several of the examples.

Claims:

- 1. Filter media comprising a fibrous material substrate incorporating an adsorbent material in particulate form wherein the adsorbent material is retained within the filter media by means of a fixing agent whereby in use shedding of the adsorbent material therefrom is inhibited.
- 2. Filter media according to claim 1 wherein the fibrous material substrate is a non-woven fabric.
- 3. Filter media according to claim 1 or claim 2 wherein 10 the adsorbent material is retained in the filter media by means of fixing agent comprising a latex binder.
 - 4. Filter media according to claim 1 or claim 2 wherein the adsorbent material is retained in the filter media by means of fixing agent comprising a film former.
- 5. Filter media according to claim 1 or claim 2 wherein the adsorbent material is introduced to the fibrous material for retention therein by treating the fibrous material substrate with a treatment liquor containing fixing agents including a binder and a film former, and said adsorbent material.
 - 6. Filter media according to claim 5 wherein the treatment liquor is applied to the fibrous material substrate by at least one of the following treatments; saturation by soaking, impregnation by immersion in the treatment liquor, forced impregnation by application of the liquor under pressure, pouring of the treatment liquor over the fibrous material, and spraying.

WO 98/30326 PCT/GB98/00001

- 7. Filter media according to claim 2 wherein at least one additional fluid-permeable fibrous material is laid over the non-woven fabric.
- 8. Filter media according to claim 7 wherein the or 5 each additional fluid permeable fibrous material is of a different structure to the non-woven fabric.
 - 9. Filter media according to claim 7 wherein the or each additional fluid-permeable fibrous material has been separately pre-treated to incorporate an adsorbent material differing from that incorporated in the non-woven fabric.
 - 10. Filter media according to claim 9 wherein the additional fluid-permeable fibrous material is also a non-woven fabric.
- 11. Filter media according to claim 8, or claim 9 in the 15 form of a laminate.
 - 12. Filter media according to claim 1 in the form a flat flexible sheet capable of conforming to a surface e.g. for use in a garment.
- 13. Filter media according to claim 1 in the form of a 20 scroll.
 - 14. Filter media according to claim 1 in the form of a wadding.
 - 15. Filter media according to claim 2 wherein the non-woven fabric is a dry-laid fabric.
- 25 **16.** A process for forming filter media comprising providing a fluid-permeable carrier substrate containing fibres which are selected from fibres capable of withstanding

prolonged contact with a fluid to be filtered, treating the carrier substrate to introduce fixing agents including a binder and a film-former, and particulate adsorbent material(s) to the carrier substrate, wherein the amount of fixing agents is just sufficient to retain the adsorbent material(s) in the carrier substrate whereby shedding of the adsorbent therefrom is substantially prevented.

- 17. A process for forming filter media wherein the filter media is formed by selecting a fluid permeable carrier substrate consisting essentially of fibres and impregnating the substrate with a liquid containing an adsorbent material in particulate form and fixing agents for promoting particle retention in the substrate without occluding the permeability of the substrate, separating the impregnated substrate and the liquid after a predetermined period sufficient to obtain retention of the particulate adsorbent material in the substrate.
- 18. A process according to claim 16 or claim 17 wherein the fluid-permeable carrier substrate is treated with a liquid 20 containing a latex binder.
 - 19. A process according to claim 18 wherein the latex binder is selected from acrylics, styrene butadiene and natural rubber based binders.
- 20. A process according to claim 16 or claim 17 wherein 25 the liquid also contains a film former.
 - 21. A process according to claim 16 or claim 17 wherein the liquid also contains an anti-foaming agent.
 - 22. A process according to claim 20 wherein the film former is selected from polyvinyl alcohol, polyvinyl

alcohol/vinyl acetate copolymers, quaternary ammonium salts of polyvinyl pyrolidine/vinyl acetate copolymer.

- 23. A process according to claim 16 or claim 17 wherein the adsorbent material(s) comprise at least one of activated carbon, zeolites, inorganic compounds such as silica and metal oxides e.g. of titanium (TiO_2) , zinc (ZnO), aluminium in crystalline or micro-crystalline or fine particulate form and mixtures of such adsorbents.
- 24. A process according to claim 16 or claim 17 wherein 10 the particle size of the adsorbent material(s) is controlled to lie within the range of from about 0.1 to about 300 microns.
 - 25. A process according to claim 16 or claim 17 wherein the particle size of the adsorbent material(s) is controlled so as not to exceed 75 microns.

15

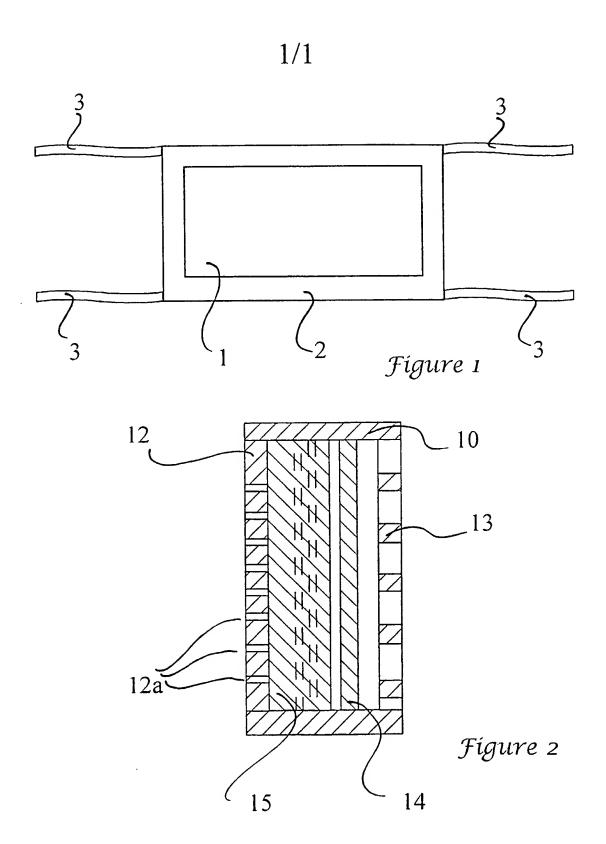
- 26. A process according to claim 16 or claim 17 wherein the particle size of the adsorbent material(s) is controlled so as not to be less than about 1 micron.
- 27. A process according to claim 16 or claim 17 wherein 20 the particle size of the adsorbent material(s) is controlled so as not to be less than about 25 microns.
 - 28. A process according to claim 16 or claim 17 wherein the carrier substrate is treated by at least one one of the following treatments; soaking, impregnation by immersion in the treatment liquor, forced impregnation by application of the liquor under pressure, pouring of the treatment liquor over the fibrous material, and spraying.
 - 29. A process according to claim 16 or claim 17 wherein

the fibres are selected from polyesters, polyamides, nylon, polyvinyl alcohol fibres, and rayon viscose fibres.

- 30. A process according to claim 16 or claim 17 wherein fibres are between 0.5 dtex and 6 dtex.
- 5 **31.** A process according to claim 16 or claim 17 wherein fibres are between 1 dtex and to 3 dtex.
 - 32. A process according to claim 16 or claim 17 wherein the carrier substrate comprises a non-woven fabric obtained by a carding process or a fibre entanglement process.
- 10 **33.** An article of protective apparel comprising filter media according to any one of claims 1 to 15 or filter media obtained by a process according to any one of claims 16 to 32.
 - 34. A filter cartridge comprising filter media according to any one of claims 1 to 15 or filter media obtained by a process according to any one of claims 16 to 32.

15

- 35. A filter element comprising a fibrous wadding or scrolled fibrous sheet incorporating an adsorbent material in particulate form which is retained therein by means of a latex binder and a film former present in sufficient quantities to avoid shedding of the particulate adsorbent(s) therefrom but permitting the fibrous wadding or sheet fabric to retain a high level of fluid permeability.
- 36. A conformable fibrous sheet incorporating an adsorbent material in particulate form which is retained therein by means of a latex binder and a film former present in sufficient quantities to avoid shedding of the particulate adsorbent(s) therefrom but permitting the fibrous sheet to retain a high level of fluid permeability.



INTERNATIONAL SEARCH REPORT

PCT/GB 98/00001

A. CLASSIFI	CATION OF SUBJECT	MATTER
IPC 6	CATION OF SUBJECT B01J20/28	B01D39/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUM	ENTS CONSIDERED TO BE RELEVANT	
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Х	US 5 124 177 A (KASMARK) 23 June 1992	1-3,6-8, 10,14, 15, 17-19, 23,24, 28,29
A	see column 2, line 10-26 see column 5, line 40 - column 6, line 48 see column 12, line 1 - column 13, line 6 see column 17, line 43 - column 18, line 28	35
X	CA 1 207 736 A (CANADA MAJESTY IN RIGHT OF CANADA) 15 July 1986	1,4-6, 16-20, 23-25, 28,35,36
	see page 11-13; claims 1-12	20,00,00
	-/	

X Further documents are listed in the continuation of box C.	X Patent family members are listed in annex.
Special categories of cited documents: A* document defining the general state of the art which is not considered to be of particular relevance E* earlier document but published on or after the international filling date L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) O* document referring to an oral disclosure, use, exhibition or other means P* document published prior to the international filling date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention." "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone. "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family
Date of the actual completion of theinternational search 28 April 1998	Date of mailing of the international search report $19/05/1998$
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Wendling, J-P

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Intern. Ial Application No
PCT/GB 98/0001

		PCT/GB 98/00001
	ation) DOCUMENTS CONSIDERED TO BE RELEVANT	
Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Ą	GB 896 345 A (AMERICAN AIR FILTER) 16 May 1962	1,6, 17-19, 23,28,35
	see the whole document	25,25,55
A	EP 0 188 182 A (GEDEVELOP) 23 July 1986 see page 3, column 4, line 47 - page 4, column 5, line 32	1,4
A	PATENT ABSTRACTS OF JAPAN vol. 016, no. 061 (C-0910), 17 February 1992 & JP 03 258319 A (KURARAY CHEM CORP), 18 November 1991, see abstract	1
A	PATENT ABSTRACTS OF JAPAN vol. 014, no. 275 (C-0728), 14 June 1990 & JP 02 080696 A (TOKUSHU SEISHI KK), 20 March 1990, see abstract	1
•		

BEST AVAILABLE COPY

INTERNATIONAL SEARCH REPORT

information on patent family members

Intern: al Application No PCT/GB 98/00001

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5124177 A	23-06-92	US 5338340 A	16-08-94
CA 1207736 A	15-07-86	NONE	
GB 896345 A		DE 1254127 B US 3019127 A	30-01-62
EP 188182 A	23-07-86	SE 452854 B CA 1254548 A DK 18786 A FI 855179 A JP 61179354 A SE 8500208 A US 4777069 A	21-12-87 23-05-89 18-07-86 18-07-86 12-08-86 18-07-86 11-10-88